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Residual Stress in a 3D Carbon-Carbon Composite

Prepared by

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30 September 1986

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OFFICE OF NAVAL RESEARCH
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AIR FORCE SYSTEMS COMMAND
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Residual stress is measured in a thin slice from a carbon-carbon composite with a 3D Cartesian weave. The curvature of a slice of asymmetric cross section indicates the amount of stress caused by thermal expansion anisotropy.

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FIGURES

I. INTRODUCTION

Residual stress, a well-known phenomenon in composites, is frequently caused by thermal expansion mismatch between reinforcement and matrix phases. Particularly for carbon-carbon composites, the consequences of thermal expansion can be severe. The extreme thermal expansion anisotropy of the graphite crystal perpendicular and parallel to the basal plane and the high stiffness parallel to the basal plane, can precipitate fracture in large composite structures during processing.

Given the high temperatures involved in processing, direct evidence for residual stress is difficult to obtain in such composites. Some work has been done by measuring cracking in processed cylindrical carbon-carbon composites (Ref. 1). Curvature or warping of machined bars of nonhomogeneous material is often observed when local variations of material properties create residual stress (Ref.2). This report discusses evidence for residual stress obtained by cutting thin slices from a 3D Cartesian weave carbon-carbon composite and measuring the residual curvature.

II. EXPERIMENTAL

In preparing slices of various thicknesses, we observed that some slices of less than a certain thickness would not remain straight but would warp noticeably (Fig. 1). Further investigation discovered the warping to be related to both the thickness of the slice relative to the yarn spacing and the slice's location in the bulk composite.

The material analyzed was a 2-2-3 (x-, y-, and z-directions, respectively) Cartesian weave composite, using PAN-based carbon yarn of approximately 380 GPa (55 Msi) modulus (Ref. 3). The x and y site spacings were 0.76 mm (30 mils), and the z site spacings were 0.84 mm (33 mils) (nominal values). The composite was fabricated from a 3D, carbon yarn preform by repeated densification by chemical vapor deposition and graphitization to temperatures above 2500°C. The matrix material was graphitic, and the composite had an overall methanol immersion density of 1.9×10^{-3} kg/m^3 (1.9 g/cm^3).

Slices 27 mm long, 4 mm wide, and 0.25, 0.5, and 1 mm thick were cut from the bulk composite. The length was parallel to the x-direction, and the width was parallel to the y-direction. Some of the 0.25-mm-thick slices curved markedly (Fig. 1a); for thicker slices, the curvature was less severe. From the optical micrographs (Figs. 1 b and c) of the front and back surfaces of the slices, which display the directions of fiber reinforcement in the material, one can conclude that the curvature is caused by asymmetric fiber reinforcement of opposite faces of the thin slice coupled with residual thermal stress.

Figure 2 diagrams the geometry of a unit cell. The site spacing along the thickness direction of the slice corresponds to 0.76 mm (30 mils). Thus, a slice cut thinner than that will contain only part of a unit cell through the thickness. Because the location of the slice was selected at random, the amount of x-direction reinforcing carbon fiber versus matrix material and y- and z-direction reinforcing fiber was somewhat variable in the slice.

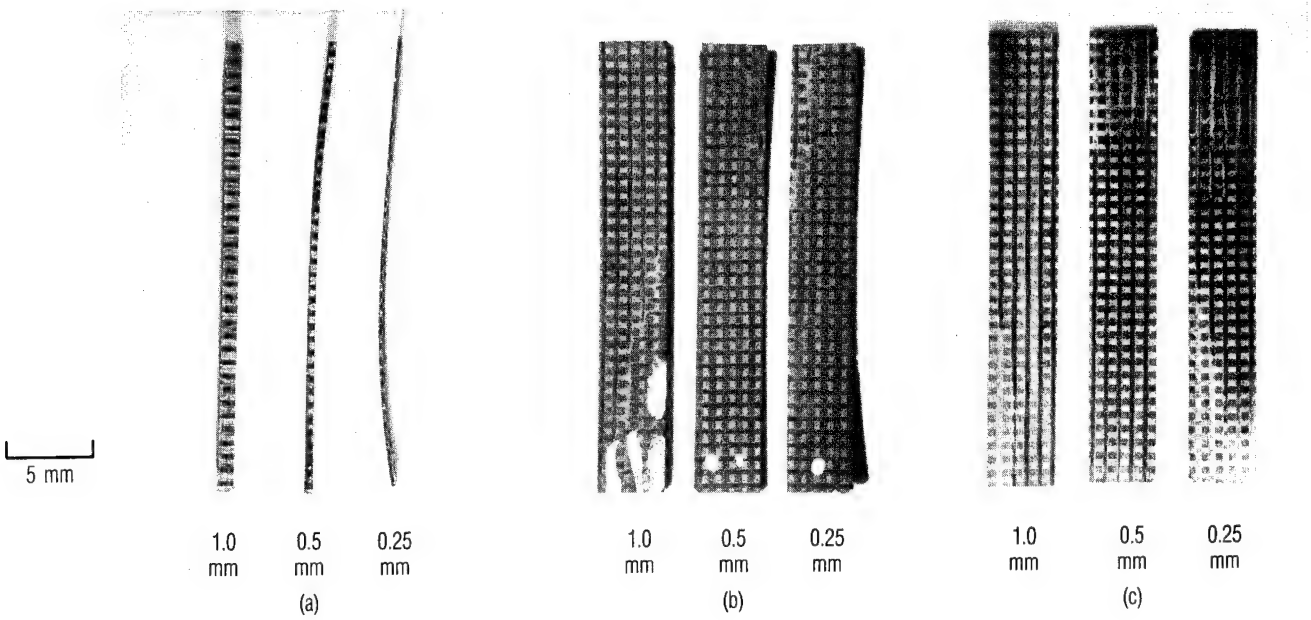

Figure 1. X-Direction Slices of 2-2-3 Composite

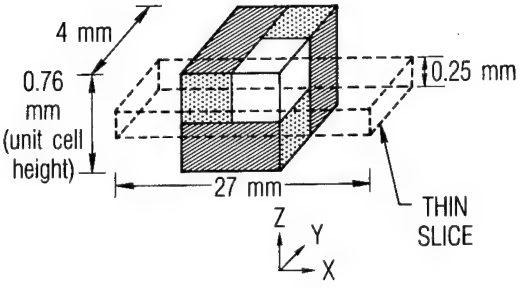

Figure 2. Geometry of Unit Cell and Thin Slice along x-Direction from 2-2-3 Composite

III. RESULTS AND DISCUSSION

In Figs. 1 b and c, x-direction reinforcing filaments in the 0.25-mm-thick slice are evident along almost the entire back surface, but none is showing on the front, although y-direction reinforcing fibers are. Matrix pockets and ends of z-direction filaments appear on both front and back faces. Because of the thermal expansion anisotropy of the fiber, the matrix and transverse directions of the y- and z-yarns in this sample tend to shrink more in cooling from elevated temperature than the longitudinal direction of the x-yarns; so the x-direction yarns are in residual compression while the matrix and transverse fibers are in residual tension. The four components--matrix and yarns in three directions--will require good bonding under conditions of local thermal expansion mismatch to retain the residual stresses.

The x-y surface of the sample is not perfectly parallel to the x-direction yarns, so the thickness of these yarns varies along the length of the sample, as the nonuniformity of the curvature along the sample illustrates. Given both this nonuniformity, which implies variation with position of the effective bending stiffness, EI, and the asymmetry of the sample with respect to the neutral surface, the relation of the composite curvature to the individual constituent properties is not simple to analyze. However, comparison in terms of a similar beam of homogeneous, isotropic material is instructive.

Assuming a thin beam, simply supported at the ends (Ref. 4), with a beam length of 27 mm, thickness of 0.25 mm, and midpoint deflection of 0.86 mm, the maximum tensile strain ε_{max} can be estimated from

$$\varepsilon_{max} = \frac{t}{2R}$$

where t is the thickness and R is the radius of curvature. Given the radius of curvature, 106 mm, which can be estimated from the deflection and length, the estimated maximum tensile strain in the material is 1.2×10^{-3}. The limited applicability of isotropic beam theory dictates the need for more

complex composite beam theory, such as the 2D laminate approach with asymmetric layup (Ref. 5); however, the estimate is reasonable by being less than the measured strain to failure of typical bulk 3D carbon-carbon composites in tensile testing (Ref. 3).

Additional evidence for residual stress is demonstrated by the bimetallic strip behavior of the sample during heating and cooling, depicted in Fig. 3 for the curved sample at different temperatures on the surface of a hot plate. On heating, the curvature decreases slightly. This is to be expected, because the thermal expansion of the fibers in the longitudinal direction is lower than that of the matrix and fibers in the transverse direction. The difference will cause the matrix and transversely oriented fibers, which are in tension, to expand more than the x-direction fibers, which are in compression. The reverse occurs on cooling the sample in liquid nitrogen (77 K), which causes a small increase in sample curvature. That the curvature returned to its initial condition at room temperature shows that the changes are reversible within the temperature range, and that, therefore, bonding integrity between the components of the composite remains good.

The change in curvature with temperature can also be related to the degree of undercooling below the stress-free temperature, at which the beam is not curved. Using a linear thermal expansion model (Ref. 6), a study of residual stress in a thin beam of copper bonded to a ceramic showed that 1/R, where R is the radius of curvature, is proportional to the degree of undercooling below the stress-free temperature, T_s, or

$$1/R = k\ (T_s - T)$$

where k is a constant and T is temperature. For a small degree of curvature, the midpoint displacement, d, is proportional to 1/R (Ref. 7). Thus, at a given temperature, T, d is proportional to $(T_s - T)$, or

$$d = k'\ (T_s - T)$$

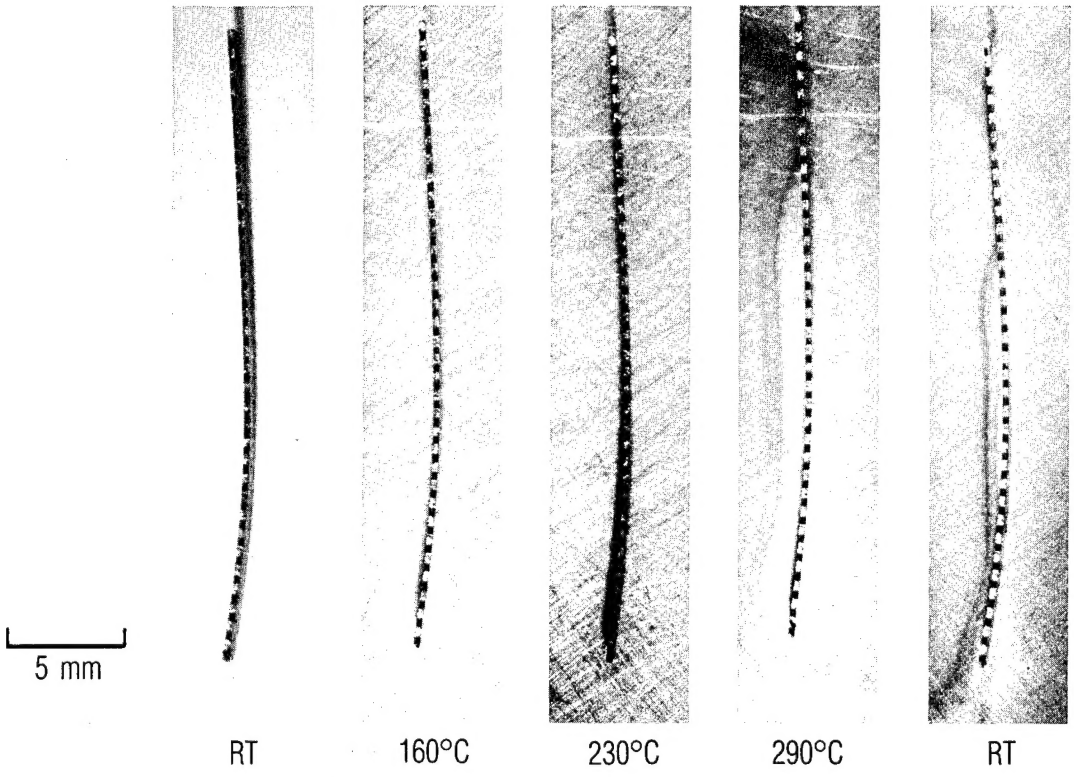

Figure 3. Changes in Curvature versus Temperature of 0.25-mm Slice of 2-2-3 Composite

where k' is a constant. From the observed values of d and T at two temperatures (e.g., room temperature and 280°C), we can solve for T_s. Thus the estimated stress-free temperature is about 1100°C (see Fig. 4), which implies that the fiber and matrix material bond well over a fairly wide temperature range and that the matrix remains sufficiently intact to retain some residual stress. The presence of residual stress and the implied large undercooling contrast with other indications that stress tends to be removed by matrix microcracking (Refs. 8 and 9) because of the large undercooling below the expected stress-free temperature, which is above 2000°C, where stress relaxation by creep becomes significant in carbon-carbon composites (Ref. 10).

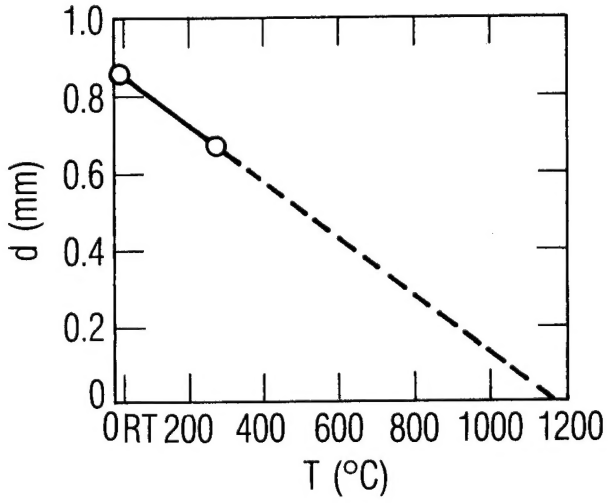

Figure 4. Measured Maximum Displacement versus Temperature

IV. CONCLUSION

In conclusion, thin slices of 3D Cartesian weave carbon-carbon composites with asymmetric cross section have been demonstrated to contain residual stresses as a result of thermal expansion anisotropy. The slices are curved, with longitudinal reinforcing filaments in residual compression and transverse fibers and matrix material in residual tension.

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